

Research Article

Dynamics of diuron and sulfentrazone formulations in soils with different textures

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INFORMATION ARTICLE

Received: April 8, 2019
Accepted: April 14, 2020

Keywords:

herbicide
leaching
commercial mix
tank mix.

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Cite this article:

Matos AKA, Carbonari CA, Brito IPFS, Gomes GLGC, Trindade MLB, Velini ED. Dynamics of diuron and sulfentrazone formulations in soils with different textures. *Planta Daninha* 2020;38:e020221524.
<https://doi.org/10.1590/S0100-83582020380100068>

Conflict of Interest:

The authors declare that there is no conflict of interest regarding the publication of this manuscript.

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HIGHLIGHTS

- The formulations did affect the leaching potential of herbicides, according to the soil properties.
- The simulation of higher rainfall volumes promoted the greater leaching of herbicides.
- The leaching potential of both formulations were reduced in medium and clayey soils.

ABSTRACT

Background: The use of commercial mix and tank mix with two or more herbicides is a widespread practice. However, despite its several possible benefits, little is known about its effect on the behavior of the molecules in the soil, which can increase or reduce the leaching potential of these molecules.

Objective: This study evaluated the leaching potential of the herbicides diuron and sulfentrazone applied, using commercial mix and tank mix, in soils with different textures.

Methods: The herbicides diuron (1,750 g ha⁻¹) and sulfentrazone (875 g ha⁻¹) were applied in three soils with different textures (clayey, medium, and sandy), using a commercial mix (Stone[®]) and a tank mix (Herburon 500 BR + Boral[®]). Subsequently, the treated soils were reconstituted in soil columns subjected to rainfall simulations with accumulated rainfall volumes of 40, 80, and 120 mm; the leached solutions were collected and analyzed by LC-MS/MS.

Results: The results indicated a low descending movement of diuron in the three soils evaluated, when applied using commercial and tank mixes with sulfentrazone. Higher rainfall volumes (80 and 120 mm) were required to increase the diuron movement (≈20% of diuron applied). Sulfentrazone showed a high leaching potential in the soils, whereas the tank mix increased the leaching potential in sandy soil with the rainfall depths of 80 and 120 mm.

Conclusions: The formulations did not affect the leaching potential of the herbicides in clayey and medium soil. However, the tank mix promoted greater leaching of the herbicides in the sandy soil when compared to the commercial mix.

1 INTRODUCTION

The current agricultural systems depend on the use of herbicides for control of weedy species and

reduce possible interferences of these species on the crop development and yield, as in the case of sugarcane crops. However, concerns about the environment fate and risk of contaminations have

emerged, since several molecules applied in this crop were found in surface water and groundwater (Dores et al., 2006; Gomes et al., 2006; Britto et al., 2012).

Residues of acetochlor, ametryn, amicarbazone, atrazine, clomazone, diclosulam, diuron, glyphosate, imazapic, isoxaflutole, pendimethalin, S-metolachlor, simazine, sulfentrazone, sulfometuron-methyl, tebuthiuron, and trifluralin were found in surface and groundwater and in semi artesian wells in several areas near sugarcane fields in Brazil (Matallo et al., 2003; Gomes et al., 2006; Armas et al., 2007; Santos et al., 2015; Santos et al., 2017). Most of these herbicides applied recurrently are persistent and have moderate to high mobility in soils; thus, they may leach from agricultural areas to groundwaters and represent risks of contamination of the environment.

Leaching is the transport of molecules – dissolved or weakly adsorbed in the soil solution – and water through the soil profile (Linde, 1994). Leaching is essential to the surface incorporation of herbicides to the soil, reaching the seed bank zone and seeds under germination process (Velini, 1992); however, excess leaching can contaminate the water table.

The sorption capacity of herbicides is the characteristic that most affect leaching; the sorption process determines the herbicide concentration available in the soil solution that can be transported (Oliveira and Brighenti, 2011). These processes depend on soil physical-chemical properties, climate conditions, and herbicide characteristics.

Organic matter is the factor that affect the most the sorption of non-ionic or cationic herbicides in tropical soils (Prata and Lavorenti, 2000). In the case of weak acids, the product availability in the soil vary according to the soil pH due to its constant dissociation (pKa). When the soil pH is lower than the pKa, its neutral form predominates; and when the pH is higher than the pKa, the anionic form predominates (Grey et al., 2000).

The application of commercial or tank mixes with two or more herbicides is a widespread practice of weed management. The increase in control spectrum, assistance in management of resistant species, and increase of residual period, which are due to the different characteristics of the products, are among the advantages described for association of herbicides (Qureshi and Vanden Born, 1979; Riley and Bradley, 2014).

Commercial mixes with the herbicides diuron and sulfentrazone were introduced to the Brazilian market in the last few years to control mono and dicotyledonous weeds in pre and/or post-emergence, in sugarcane crops. Diuron (N'-(3,4-dichlorophenyl)-N,N-dimethylurea) is a herbicide of the phenylureas group; it consists of non-ionizable molecules whose main characteristics is low solubility in water (42 mg L⁻¹ to 25 °C), moderate octanol-water partition coefficient (Kow of 589), and high organic carbon/water partition coefficient (Koc of 480 mL g⁻¹). It is strongly adsorbed to the soil organic and clay fractions (Shaner, 2014), and is a persistent herbicide, with half-life in the soil of 90 days up to one year. It is moderately mobile, tend to leachate, mainly in the soils with low organic matter contents; in this condition, the desorption process is more intense than the sorption (Liu et al., 2010).

Sulfentrazone (N'[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3methyl-5-oxo-1H-1,2,4-triazol-1-yl]phenyl]methanesulfonamide) is a herbicide of the aril triazolinone group; it is a weak acid (pKa 6.56), with low Kow (9.8 at pH 7) and increasing solubility in water as the pH is increased –110 mg L⁻¹ at pH 6; 780 mg L⁻¹ at pH 7; and 1,600 mg L⁻¹ at pH 7.5, at 25 °C (Shaner, 2014). Once in the soil, sulfentrazone presents mobility and high persistence (half-life of 121 to 302 days under field conditions), with low sorption capacity (Kd < 1.0 mL g⁻¹ and Koc of 43 mL g⁻¹) (Shaner, 2014); thus, it presents high risk of contamination of underground waters, and is classified as highly leachable according to the GUS index (6.16) (PPDB, 2018). The mobility of this herbicide is related to the soil pH and organic matter content, although the clay fraction may also affect it (Ohmes and Muller, 2007; Szmigielski et al., 2012).

The antagonism, synergism, and efficiency of commercial and tank mixes have been evaluated in several studies (Petter et al., 2012; Chhokar et al., 2013; Riley and Bradley, 2014; El-Nahal; Hamdona, 2017; Rakes et al., 2017; Reis et al., 2017). However, despite the several benefits of this practice, little is known about its effects on the leaching potential of molecules.

Several interactions can affect the dynamics of herbicides in the soil when applied mixed, including some that are related to competition for sorption sites, which can increase or reduce the leaching potential of these molecules (Farenhorst and Bowman, 1998; Farenhorst and Prokopowich, 2003; Shariff, 2012). In this context, the objective of this study was to evaluate the leaching potential of the herbicides

diuron and sulfentrazone applied, using commercial mix and tank mix, in soils with different textures.

2 MATERIAL AND METHODS

The experiments were conducted under controlled condition in a laboratory at the Department of Plant Protection of São Paulo State University, Botucatu campus, Brazil. The experiments evaluated the leaching potential of formulations with the herbicides diuron and sulfentrazone applied on soil columns. Soils with clayey, medium, and sandy textures were used in each experiment. A completely randomized design with four replications was used, in a 3X2 factorial arrangement consisted of 3 accumulated rainfall depths (40, 80, and 120 mm) and two herbicides formulations (commercial mix, and tank mix), totaling six treatments.

The soils were selected according to their texture and organic matter content. They were collected from

the 0-10 cm layer, dried in a forced-air circulation oven at 40 °C, and sieved in 2 mm mesh sieves. Aliquots of these soils were sent for laboratorial analysis for physical-chemical characterization; the results are described in Table 1.

The experimental units consisted of polypropylene columns of 11.5 cm height and 2.9 cm diameter, with rough internal walls to avoid the lateral slides. The lower 5 mm of the columns were filled with washed sand and a glass fiber paper filter (pore size 0.7 µm, Millipore®, EMD Millipore Corporation, Billerica, MA, USA) used to avoid soil losses during the experiment; the rest of the column was filled with 10 cm of non-treated soil and 1 cm of soil treated with the herbicide formulations (Figure 1A). The soils were placed in 907.46 cm² trays, making a layer of 1 cm height for the application of the treatments. The formulations of the herbicides diuron (1,750 g a.i. ha⁻¹) and sulfentrazone (875 g a.i. ha⁻¹) were applied to the soil

Table 1 - Physical-chemical characteristics of the soils used in the experiment

Soil	Sand	Clay	Silt	Texture	pH	OM	P _{resin}	
	(g kg ⁻¹)				(CaCl ₂)	(g dm ⁻³)	(mg dm ⁻³)	
1	246	553	201	Clayey	4.8	23	16	
2	632	281	87	Medium	5.3	11	18	
3	958	29	13	Sandy	7.0	6	39	

Soil	Al ³⁺	H+Al ³⁺	K ⁺	Ca ²⁺	Mg ²⁺	SB	CEC
	(mmol _c dm ⁻³)						
1	2	48	1.3	23	10	34	83
2	1	39	0.8	21	11	32	71
3	0	6	1.4	24	12	38	44

Soil	BS	S	B	Cu	Fe	Mn	Zn
	(mg dm ⁻³)						
1	41	56	0.26	9.7	6	25.2	1.3
2	46	38	0.21	0.7	25	0.7	0.3
3	86	43	0.11	0.4	13	2.7	0.0

OM = organic matter; SB = sum of bases; CEC = cation exchange capacity; BS = bases saturation (%).

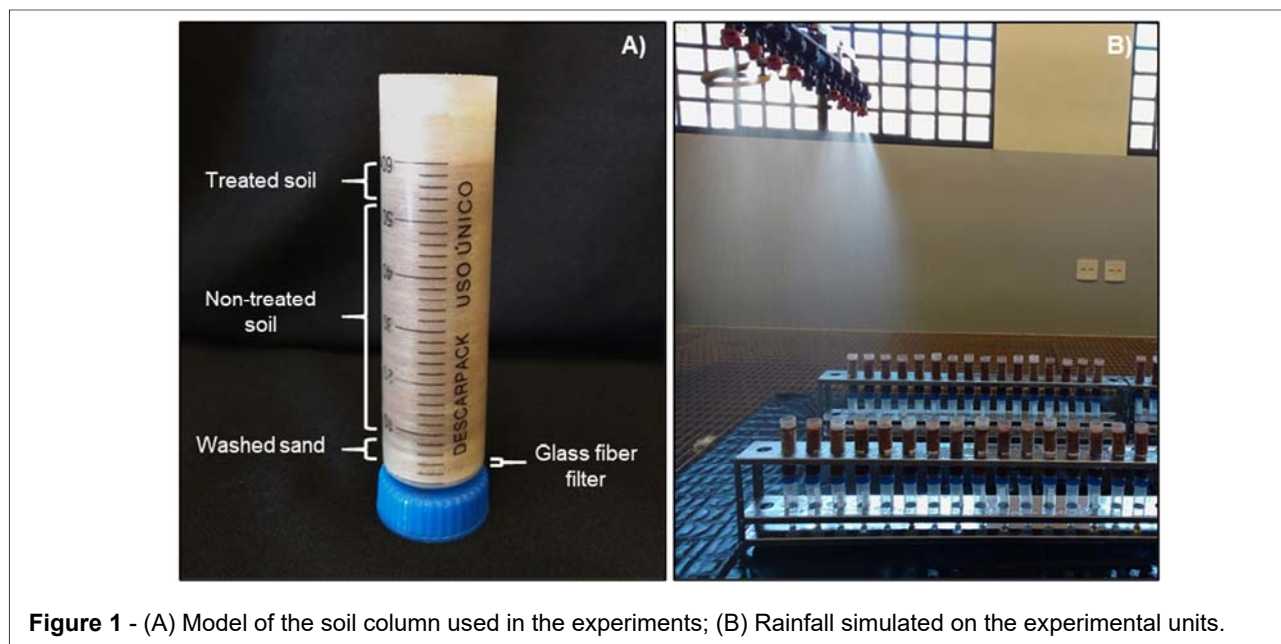


Figure 1 - (A) Model of the soil column used in the experiments; (B) Rainfall simulated on the experimental units.

surface, using a commercial mix (Stone[®] SC, FMC Corporation) and a tank mix (Herburon 500 BR, ADAMA Brazil S.A.+ Boral[®] 500 SC, FMC Corporation).

A spraying automated system with speed and pressure control was used for the applications. The spraying device consisted of a spray boom with four flat-fan nozzles XR110.02VS (Teejet[®] Technologies, Wheaton, IL, USA) spaced 0.5 m apart and placed at 0.5 m above the trays surface. The system was operated at a traveling speed of 1.0 ms⁻¹, with spray volume of 200 L ha⁻¹ and constant pressure of 193.0 kPa. The environmental conditions at the time of application were monitored and showed an air temperature of 27 °C and relative air humidity of 50%.

The soils samples were homogenized after the application and a 1-centimeter layer of each soil was reconstituted in the columns filled with non-treated soil. The experimental units were subjected to cumulative rainfalls of 40, 80 and, 120 mm at 24 h after the application.

The rainfall simulation was performed using the same structure previously described with rainfall simulator boom coupled to the spraying device, with eight nozzles DG9505EVS (Teejet[®], Wheaton, USA), spaced 5.0 cm apart, positioned at 1.4 m above the soil columns and set to achieve the different rainfall depths tested (Figure 1B). After the rainfall simulations, the percolated soil solutions were collected, measured, filtered in porous membrane of PVDF 0.45 µm (Millex-HV[®], EMD Millipore Corporation, Billerica, MA, USA), and stored in vials (2 mL) for later chromatographic detection of herbicides.

The herbicides concentrations in the percolated soil solutions were assessed using a high-performance liquid chromatograph (Prominence UFLC, Shimadzu, Kyoto, Japan), coupled to a hybrid triple quadrupole mass spectrometer (AB Sciex Triple Quad 4500; Sciex, Framingham, USA), LC-MS/MS system. The chromatographic conditions to quantify the compounds were performed in the gradient mode, with the solvents methanol and 0.5% acetic acid (Table 2). The electrospray ionization (ESI) source chosen allowed the detection and separation of compounds in a single run for positive and negative modes.

The total run time for the sulfentrazone and diuron herbicides was 10 minutes, with retentions time in the chromatographic column of 4.96 and 5.30 minutes; the analytical curves were $y=8.33x+5.24$ and

Table 2 - Chromatographic conditions used for the compounds quantification

Analytical column	C18 Synergi 2.5 µ Hydro RP 100 Å
Volume of injection	20 µL
Mobile phase (pH 7.0)	Phase A (FA) = 0.5% acetic acid in water Phase B (FB) = 0.5% acetic acid in methanol
Gradient mode	0 – 1 minutes = 50% FB and 50% FA 1 – 3 minutes = 95% FB and 5% FA 3 – 6 minutes = 95% FB and 5% FA 6 – 8 minutes = 50% FB and 50% FA 8 – 10 minutes = 50% FB and 50% FA
Flow rate	0.60 mL min ⁻¹
Oven temperature	40 °C

$y=3.743x+4.693$, with coefficient of determination of $R^2=0.9935$ and $R^2=0.9918$, respectively.

The leaching of the different formulations of diuron and sulfentrazone was expressed as a percentage of the total applied to each soil tested. The leaching was estimated by analysis of herbicide mass balance for each formulation, the quantity applied was determined by analysis of herbicides in the soils, weighted by the tray area units and amount of soil (mg g⁻¹), and the quantity of compounds in the solution percolated through the columns was determined for each rainfall depth tested by analysis of compounds in the solutions collected after the rainfall simulation.

The data were subjected to analysis of variance by the F test (ANOVA, $p \leq 0.05$), and the means were compared by the Tukey test at $p \leq 0.05$. Analyses of linear correlation were carried out for the three soils, considering the leaching of the herbicides diuron and sulfentrazone as a function of the different formulations applied.

3 RESULTS AND DISCUSSION

The dynamics of the herbicides indicated a low descending movement of diuron in the three soils evaluated, when applied using commercial and tank mixes with sulfentrazone. In the soil with clayey texture, high percentages of diuron were leached only after the simulation of accumulated rainfall depths of 80 and 120 mm, showing that the rainfall depth of 40 mm was not enough to drag the herbicide molecules throughout the soil profile. The percentages found in the leached solutions in the rainfall depth of 40 mm were lower than 0.44% of the total diuron applied (Table 3), whereas the highest rainfall depths resulted in percentages of 5.96% (80 mm) and 16.65% (120 mm). Similarly, the soil with medium texture showed increases in movement of diuron as the simulated rainfall depths were increased. Means of

12.84% of the total commercial mix, and 11.58% of the total tank mix applied were leached after 120 mm of rainfall.

In the soil with sandy texture, the rainfall depth of 40 mm resulted in the movement of lower percentages of the total of diuron applied using the commercial mix (1.65%) and tank mix (3.97%), not differing from each other (Table 3). A higher movement of diuron was found when using the tank mix with simulated rainfall of 80 and 120 mm; however, the differences between the mixes were small, not enough to infer that the application of tank mix favored the leaching. Reis et al. (2017) found low mobility for diuron, applied single or combined with hexazinone and sulfometuron-methyl, in the upper soil layer (0-5 cm) of soils with sandy and clayey textures, even after a simulated rainfall depth of 192 mm, regardless of the application mode. Thus, the differences in percentages of diuron leached can be attributed to differences in soil characteristics and simulated rainfall depths.

Table 3 - Diuron leaching percentage applied in commercial mix and tank mix on soil columns after different rainfall volumes were simulated

Soil	Product	40 mm	80 mm	120 mm
Clayey	Commercial mix	0.44 Ac	5.96 Ab	16.65 Aa
	Tank mix	0.35 Ac	5.96 Ab	16.28 Aa
	F Product		0.061	
	F Rainfall depth		233.37*	
	F Product × Rainfall depth		0.033	
		CV (%)		19.86
Medium	Commercial mix	0.30 Ac	4.26 Ab	12.84 Aa
	Tank mix	0.26 Ac	4.17 Ab	11.58 Aa
	F Product		0.504	
	F Rainfall depth		114.81*	
	F Product × Rainfall depth		0.369	
		CV (%)		28.82
Sandy	Commercial mix	1.65 Ac	10.21 Bb	20.75 Ba
	Tank mix	3.97 Ac	13.31 Ab	23.88 Aa
	F Product		19.064*	
	F Rainfall depth		298.347*	
	F Product × Rainfall depth		0.167	
		CV (%)		13.00

* Means followed by the same uppercase letter in the columns or lowercase letter in the rows are not different by the Tukey's test ($p \leq 0.05$).

The adsorption capacity of non-ionic hydrophobic herbicides, such as diuron, is related to the soil organic matter content and, to a lesser extent, to the cation exchange capacity, which is strongly affected by soil organic carbon contents (Benoit et al., 2008; Liu et al., 2010; El-Nahhal et al., 2013). The hydrophobicity of diuron favors its adsorption to the soil organic fraction by more stable and higher energy bonds than those by mineral compounds (Bailey and White, 1964; Spark and Swift, 2002). Thus, the higher organic matter (SOM) contents and cation exchange capacity (CEC) of medium texture soil (SOM of 11 g dm⁻³ and CEC of

71 mmol_c dm⁻³) and clayey texture soil (SOM of 23 g dm⁻³ and CEC of 83 mmol_c dm⁻³) contributed to a higher adsorption and lower leaching of diuron when compared to the sandy soil (SOM of 6 g dm⁻³ and CEC of 44 mmol_c dm⁻³) (Table 1).

The results of sulfentrazone showed a high leaching potential in the soils evaluated for the two formulations tested (Table 4). In the clayey soil, the percentages of the herbicide in the leached solution increased as the simulated rainfall depths were increased. The rainfall depth of 40 mm resulted in the lowest movement of the herbicide throughout the soil columns (32% of the total sulfentrazone applied), whereas the rainfall depths of 80 and 120 mm resulted in leaching percentages above 60% of the total herbicide applied for both mixes. In the soil with medium texture, the simulated rainfall depth of 40 mm resulted in leaching of 42.38% and 44.41% of total sulfentrazone applied for the commercial mix and tank mix, respectively. However, the simulated rainfall depths of 80 and 120 mm resulted in increases of 66.00% to 74.37% in the percentages of sulfentrazone in the leached solutions, with no significant differences in the movement of the herbicide between these rainfall depths, and the formulations used.

Table 4 - Sulfentrazone leaching percentage applied in commercial mix and tank mix on soil columns after different rainfall volumes were simulated

Soil	Product	40 mm	80 mm	120 mm
Clayey	Commercial mix	33.37 Ac	68.01 Ab	77.82 Aa
	Tank mix	32.10 Ac	65.61 Ab	75.00 Aa
	F Product		6.904	
	F Rainfall depth		1033.702*	
	F Product × Rainfall depth		0.319	
		CV (%)		3.44
Medium	Commercial mix	42.38 Ab	66.00 Aa	71.99 Aa
	Tank mix	44.41 Ab	68.29 Aa	74.37 Aa
	F Product		1.491	
	F Rainfall depth		98.678*	
	F Product × Rainfall depth		0.003	
		CV (%)		7.32
Sandy	Commercial mix	69.01 Ba	71.01 Ba	71.19 Ba
	Tank mix	75.00 Aa	77.25 Aa	77.55 Aa
	F Product		37.699*	
	F Rainfall depth		2.214*	
	F Product × Rainfall depth		0.012*	
		CV (%)		3.36

* Means followed by the same uppercase letter in the columns or lowercase letter in the rows are not different by the Tukey's test ($p \leq 0.05$).

The application of the tank mix resulted in the highest leaching percentages of sulfentrazone than the application of the commercial mix, in the sandy soil (Table 4). However, the differences in percentages of leached sulfentrazone of each mix were not enough to infer that the application mode

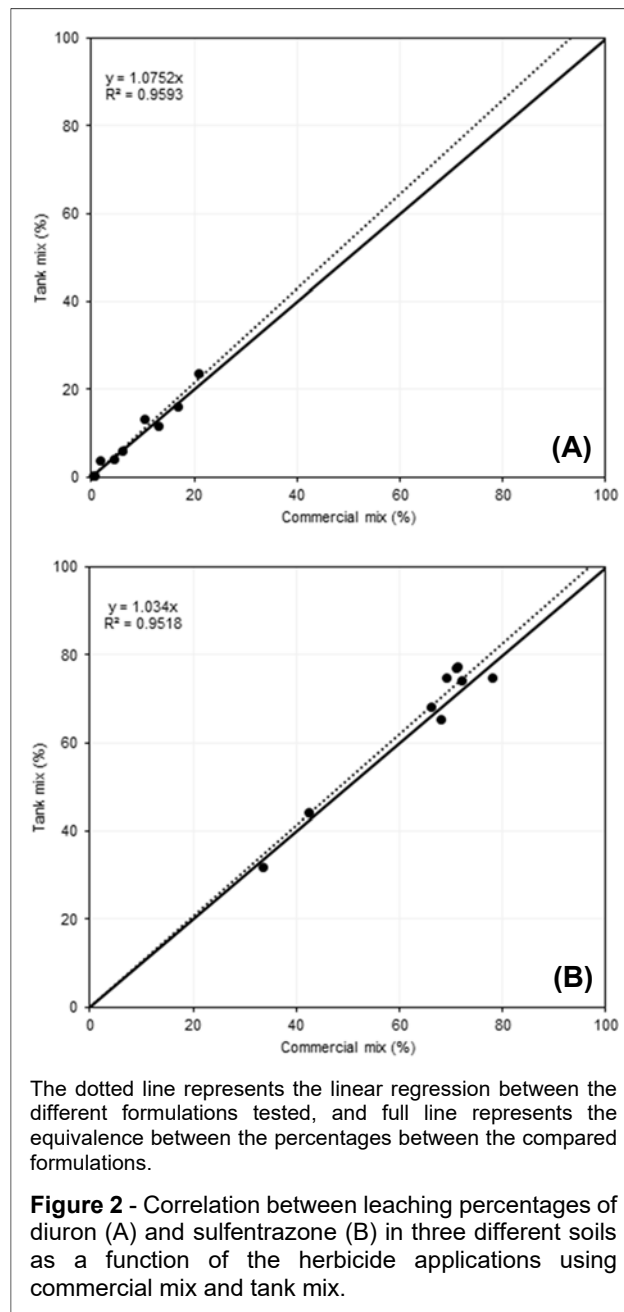
affected the movement of the herbicide. No differences were found for leaching of sulfentrazone between the simulated rainfall depths, which showed leaching percentages of 69.01% to 71.19% for the commercial mix and 75.00% to 77.55% for the tank mix.

The high clay (281 and 553 g kg⁻¹) and organic matter (11 and 23 g dm⁻³) contents of the soils with medium and clayey texture (Table 1) may explain the need for more intense rainfalls (80 and 120 mm) to increase the leaching of sulfentrazone. Contrastingly, the low clay (29 g kg⁻¹) and organic matter (6 g dm⁻³) contents of the sandy soil, combined with the high solubility (780 mg L⁻¹ at pH 7 to 25 °C) of sulfentrazone, favored the leaching of most of the herbicide applied, even after the lowest simulated rainfall depth (40 mm).

The results of this study indicate different mobility of sulfentrazone for soil physical and chemical characteristics, especially clay and organic matter contents. The sorption of sulfentrazone has positive correlation to the soil mineral and organic fractions; soils with high clay and organic matter contents favor the adsorption process, decreasing desorption and, thus, the herbicide movement (Grey et al., 1997; Passos et al., 2013). Passos et al. (2015) found that the sulfentrazone mobility is restrict to the surface layer (0-15 cm) of soils with high clay contents after a simulated rainfall depth of 60 mm; however, the herbicide is leached to higher depths in sandy soils and in soils with low organic matter contents.

Porosity is another soil characteristic that can directly affect the leaching of herbicides. Soils with sandy texture have, in general, more macropores than clayey ones; this characteristic result in higher water percolation rates and, consequently, favors the leaching of herbicides due to its higher hydrodynamic dispersity (Carter, 2000); however, micropores predominate in more clayey soils, limiting the movement of waters and herbicides. Thus, herbicides such as diuron, which have lower solubility and higher density, tend to move less in the soil than those with more soluble and lower-density molecules, such as sulfentrazone (Weber and Whitacre, 1982).

The dynamics of the two formulations with diuron and sulfentrazone were compared by fitting the results of leaching of the herbicides in the three soils studied to linear regression models, with high coefficients of determination (R²=0.95 to 0.96) (Figure 2A and B).



Considering equivalence line as a reference, the leached percentages of herbicides in the different soils were similar for the commercial mix and tank mix, indicating that the use of these formulations had no significant effect on the leaching potential of diuron and sulfentrazone. Therefore, the variations in mobility of the herbicides found in the present study can be related to the different soil characteristics and molecules that composed the mixes, and the simulated rainfall depths used.

4 CONCLUSIONS

The formulations did not affect the leaching potential of the herbicides in clayey and medium soil. However, the tank mix promoted greater leaching of the herbicides in the sandy soil when compared to the commercial mix.

5 CONTRIBUTIONS

AKAM, CAC, and EDV: designed the experiments. AKAM, IPFSB, GLGCG, and MLBT: conducted the experimental work and collected the data. CAC and EDV: contributed to the data analysis and interpreted the results. AKAM and IPFSB: wrote the manuscript under the supervision of CAC and EDV, GLGCG and MLBT.

6 ACKNOWLEDGEMENTS

The authors are grateful to the Universidade Estadual Paulista “Júlio de Mesquita Filho” UNESP, Faculdade de Ciências Agrônômicas for its facilities and support for this research.

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